

Effect of PECVD SiN_x deposition process parameters on electrical properties of SiN_x/AlGaIn/GaN structures

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Abstract

The effect of parameters of plasma enhanced chemical vapor deposition (PECVD) processes for SiN_x film fabrication on the electrical parameters of dielectric/AlGaIn/GaN structures has been studied. The effect of growing film composition, additional heterostructure surface treatment with nitrogen plasma before dielectric deposition and HF biasing during treatment on the parameters of the C–V and I–V curves of SiN_x/AlGaIn/GaN structures has been analyzed. We show that films with nitrogen to silicon concentration ratios of 60 and 40% and a high oxygen content exhibit a decrease in the positive fixed charge in the structures although the I–V curves of the structures exhibit current oscillations. Information has been reported on the effect of PECVD process mode on current oscillation parameters, e.g. period and amplitude, and length of I–V curve section in which oscillations occur. Possible explanation of these oscillations has been suggested. Additional nitrogen plasma treatment of heterostructure surface before monosilane supply to the chamber changes the magnitude and sign of fixed charge and reduces the free carrier concentration in the 2D gas channel of SiN_x/AlGaIn/GaN heterostructures. Experimental evidence has been provided for the effect of PECVD process parameters and surface preparation on the electrical parameters of the heterostructures grown.

Keywords

heterostructure, passivating coating, PECVD deposition technique, C–V curve, HEMT, I–V curve, piezoelectric charge, 2DEG.

1. Introduction

Dielectric films used as passivating coatings in high electron mobility transistors (HEMT) based on nitride compounds are important for achieving the required properties of the devices, e.g. low leakage current, and for the elimination of the detrimental current collapse phenomenon. Passivating coatings are usually formed from high dielectric permeability films, the industrial film deposition method for nitride device fabrication being plasma enhanced chemical vapor deposition (PECVD) and the dielectrics being SiN_x and SiON films [1, 2].

Existing research data show that positive fixed charge may form in dielectric/AlGaIn/GaN structures synthesized using plasmachemical processes with almost any known precursors [3]. In turn the formation of the positive charge shifts the C–V curves of the structures towards negative voltages [3–6]. Experimental data have shown that the major origin of the positive charge in SiN_x film structures is the large piezoelectric charge generated by changes in the elastic stresses in the AlGaIn layer as a result of dielectric film deposition. It was shown [3] that the synthesized dielectric film may have internal mechanical stresses of about 300–600 MPa which is an order

of magnitude lower than the stress in the AlGaIn buffer layer caused by the lattice mismatch between buffer layer and the underlying GaN layer. High resolution X-ray diffraction showed [7] that PECVD of a 40 nm SiN_x film increases the tensile stress in the AlGaIn layer by 15%, the carrier concentration in the 2D electron gas (2DEG) channel increasing by 9,5%. The positive fixed charge estimated, e.g. for SiN_x/AlGaIn structures with a 0.26 molar content of aluminum and a 20 nm barrier layer was

$$Q_{\text{const}} = (2.76 \pm 2.81) \cdot 10^{-2} \text{ K/m}^2.$$

However fixed charge decreased with an increase in the molar content of aluminum. It was shown [8, 9] that the mechanical properties of the protective film may also affect the elastic stresses produced in the AlGaIn layer upon the formation of dielectric/AlGaIn/GaN structures.

Electric states generated at the dielectric/semiconductor interface during the formation of protective coatings are of a great interest. Parameters of film/heterostructure surface interface were analyzed [1–10], including the possibility of the formation of donor-like trapping centers due to the impact of plasma ions during PECVD processes. It was reported [8] that there are but a few trapping states in the dielectric film bulk: these states locate very close to the dielectric/semiconductor interface. The composition of the plasma was also shown to play an important role in the process. For example, it was shown [12] that deep traps with $\Delta E > 0.657$ eV are generated during the synthesis of SiO₂ and SiON films with N₂O reaction gas, whereas SiN_x film deposition with NH₃ plasma generates defects with a high density of shallow traps having the energy $0.46 < \Delta E < 0.57$ eV due to AlGaIn surface saturation with N₂ vacancies. However analysis of the Auger spectra of dielectric/AlGaIn/GaN structures [9] suggested that oxygen plays the key role in the formation of fixed charge in SiON/AlGaIn/GaN structures. Oxygen excess in the film changes its properties, leading in turn to stress reduction in the AlGaIn layer and reducing the piezoelectric charge in the AlGaIn/GaN system.

Thus there are plenty of works dealing with analysis of PECVD dielectric films for the synthesis of protective coatings in AlGaIn/GaN structures but none of them clarify which PECVD process parameters determine specific properties of the synthesized structures. There are several works dealing with the effect of nitrogen plasma treatment of the AlGaIn/GaN structure before SiN_x coating deposition [13–15], but they do not analyze the effect of ion bombardment parameters on the change in the electrical properties of the SiN_x/AlGaIn/GaN structures. Though, this analysis would be quite helpful in understanding the physics of the processes involved and for the optimization of HEMT device technologies.

Below we consider further optimization possibilities of SiN_x film PECVD processes. To this end we studied how the electrical parameters of SiN_x/AlGaIn/GaN structures depend on the following process features: synthesized film composition, heterostructure surface pretreatment

with nitrogen plasma before dielectric deposition and HF bias during this plasma treatment.

2. Experimental

The films were PECVD synthesized in a Plasmalab System 100 ICP180 plant of Oxford Instruments Plasma Technology with an ICP source and a substrate HF biasing unit allowing control of HF plasma excitation power and independent control of ion beam acceleration toward the substrate. This feature is provided by a 13.56 MHz HF generator connected to the ICP source for producing dense plasma (the HF power was 1200 W) and another HF generator working at the same frequency connected to the substrate holder. The interaction between the HF power supplied to the substrate and plasma on the substrate surface generates a negative DC potential which is used for independent control of constant bias at the substrate relative to the unit enclosure. It is well-known that for the PECVD technique the plasma produced in the chamber has a positive potential relative to the chamber walls. There are estimates [10] that this potential is 10 to 40 V, and the same is the potential of the plasma relative to the substrate. If the substrate is biased from an external source, its potential is equal to that of the chamber walls (zero). Therefore the plasma, even without HF biasing of the substrate, delivers a flux of substrate-bombarding positive ions (nitrogen, silicon, oxygen and respective radicals).

The high dissociation rate developed by the ICP source allows using nitrogen instead of NH₃ as a precursor for SiN_x film deposition thus having a lower hydrogen concentration in the working chamber and hence in the growing film. Nitrogen is supplied to the ICP source chamber and monosilane is fed through the distribution ring located near the substrate holder so that to bring the SiH₄ decomposition products as close as possible to the substrate. The nitrogen flowrate was 15.8 cm³/min and the monosilane flowrate was 11.2 cm³/min. The working pressure in the chamber (1.33 Pa) was maintained by automatic vacuum valve opening control. Every deposition process was preceded by different-duration nitrogen plasma treatment of substrate surface in the same mode without monosilane supply. Monosilane supply to the chamber was turned on after treatment completion. The substrate temperature was maintained at 200 °C with an electric heater in the substrate holder, and the helium flowrate between the substrate and the substrate holder was maintained at 10 cm³/min.

The raw specimens were 52 mm diameter MOCVD AlGaIn/GaN heterostructures on sapphire substrates with a 20–25 nm AlGaIn layer and an Al molar content of 0.26–0.28. Before loading into the chamber the heterostructures were subjected to 80 °C heat treatment for 90 s in a dimethyl jet at a 80 atm pressure followed by a 30 s treatment in an isopropyl alcohol jet at 30 °C and nitrogen gas drying of the specimens. The thicknesses and

refraction indices of the dielectric films were assessed against reference silicon specimens subjected to the same plasmochemical treatment. The thicknesses and refraction indices of the films were measured at five points with a Gartner L1165300 ellipsometer at a 632.8 laser wavelength. The reference specimen thickness scatter was within 0.11% and the refraction index scatter was within 0,03%.

The capacity was measured with a Semiconductor Measurement System MDC CSM/Win instrument and the C–V curves were taken at $f = 1$ MHz and 10 kHz. The curves were recorded with planar probes, the first 0.005 cm² mercury probe the second contact probe being ring-shaped with an area 38 time that of the measuring probe. The capacity recovery hysteresis was assessed by taking C–V curves in different control voltage ranges. The I–V curves were taken simultaneously at different sweep rates (sweep cycle duration 0.1 to 0.9 s). The C–V curves were recorded using a (C_s – R_s) equivalent serial circuit. The carrier concentration in 2DEG was measured using two methods: from the initial HF currents measured using a contactless nondestructive technique on

a LEI-1600 Mobility Systems instrument (in 1/cm²) and from C–V curves ($\Delta C/\Delta V$) in cm⁻³. The depth of the free carrier concentration peak, i.e., the 2DEG depth, was also determined. The concentration and depth were calculated from the C–V curves for dielectric/AlGaIn/GaN structures and AlGaIn/GaN heterostructures after chemical stripping of the dielectric film.

3. Results and discussion

Study of the role of nitrogen concentration in SiN_x films for different PECVD modes showed that an increase in the nitrogen concentration reduces the positive fixed charge in the SiN_x/AlGaIn/GaN structures, reduces the refraction index of the films and increases their dielectric permeability at a ~2% oxygen concentration (Table 1).

The I–V curves of the dielectric/AlGaIn/GaN structures exhibit current oscillations at nitrogen to silicon concentration ratios of 60 and 40%.

As a rule the I–V curves of AlGaIn/GaN heterostructure Schottky diodes are represented as asymmetrical

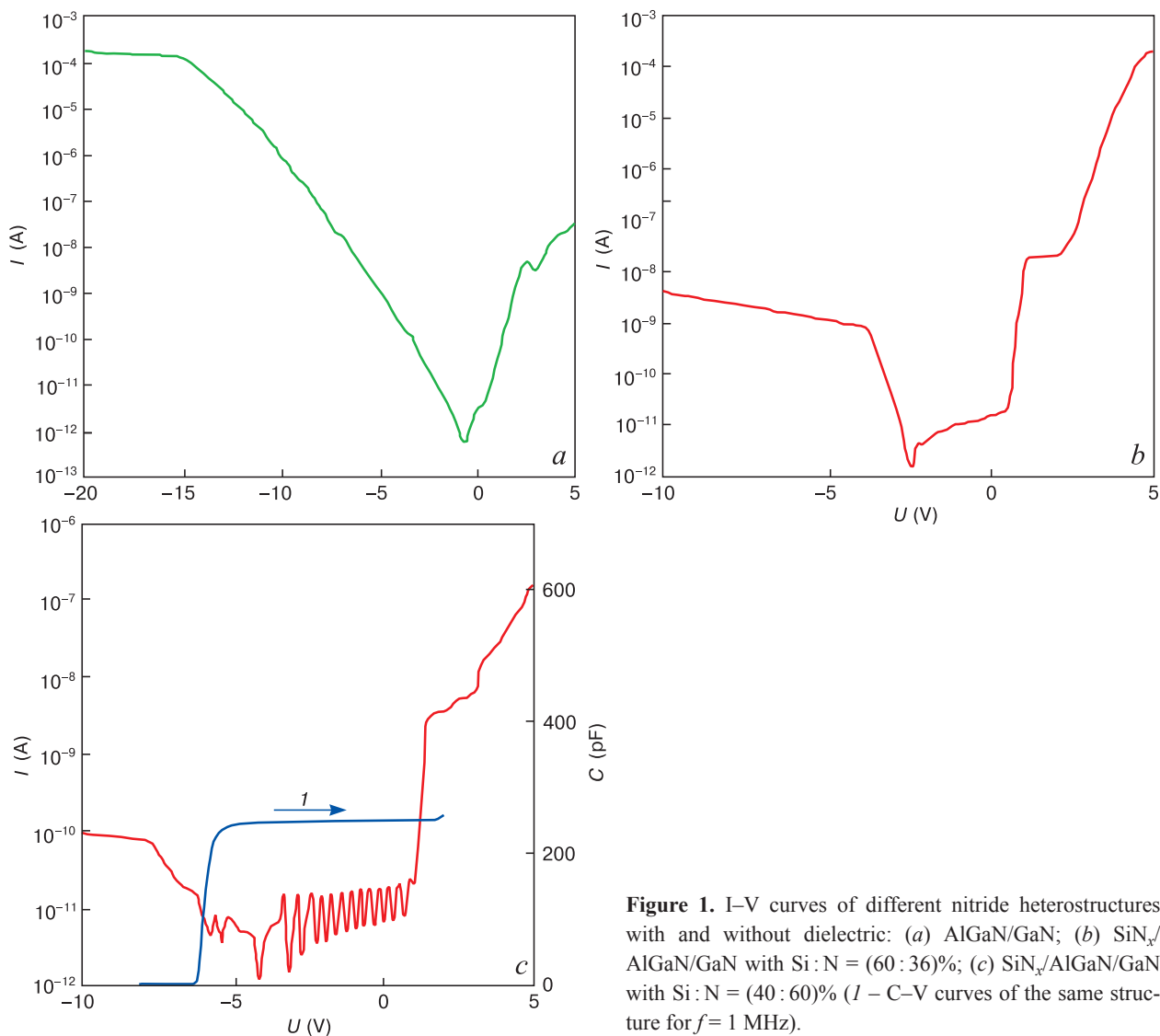


Figure 1. I–V curves of different nitride heterostructures with and without dielectric: (a) AlGaIn/GaN; (b) SiN_x/AlGaIn/GaN with Si : N = (60 : 36)%; (c) SiN_x/AlGaIn/GaN with Si : N = (40 : 60)% (I – C–V curves of the same structure for $f = 1$ MHz).

Table 1. Elemental contents in SiN_x films and their effect on parameters and cutoff voltage of dielectric/AlGaIn/GaN structures

Specimen No.	Elemental contents (%)			Refraction index	Dielectric permeability	Cutoff voltage (V)
	N	Si	O			
1	36	63	<2	2.72	6.2–6.5	–15 ÷ –17
2	60	40	<2	1.78	7	–5 ÷ –6
3	55–56	42	5–8	1.85–1.92	–	–4 ÷ –6
4	> 60	–	–	1.65–1.66	7.5–7.6	–2.5 ÷ –4

Notes. The films were deposited at $T = 400$ °C. The dielectric permeability was assessed based on AlGaIn layer capacity calculated from measured dielectric/AlGaIn/GaN structure capacity.

branches located in the positive half of the Y axis for either positive or negative control voltage [16, 17]. The current decreases dramatically as the control voltage tends to zero, the curve has a “beak” shape (Fig. 1 *a*). For the dielectric/AlGaIn/GaN structures in question the minimum currents usually shift relative toward zero control voltage and form the so-called “shoulder”. Figure 1 *b* shows the I – V curves (Table 1) for silicon rich SiN_x dielectric. The I – V curves of dielectric/AlGaIn/GaN structure containing 60% nitrogen and 40% silicon exhibited current oscillations (Fig. 1 *c*). The control voltage range in which the I – V curves had current oscillations usually coincides with the voltage range in the C – V curves of the same structures where the measured capacity C is almost constant (Fig. 1 *c*). Although the current is low in the oscillation section of the I – V curves (10^{-10} – 10^{-11} A), the oscillations have a clear shape as well as definite amplitude and period which depend on the type of structures and sweep rate.

Similar current oscillations were observed earlier in the I – V curves of dielectric/AlGaIn/GaN structures where the dielectric was a SiON film [9].

The effect of direct nitrogen plasma treatment with different duration on the electrical parameters of SiN_x/AlGaIn/GaN structures proved to be as follows:

- even a short nitrogen plasma treatment (25 s) before monosilane supply produces little changes in the measured capacity for direct control voltage sweep and almost completely eliminates positive fixed charge in the structures. U_{cutoff} shifts to zero (Fig. 2, Curves 1–3);

- an increase in the plasma treatment duration further slightly reduces the measured capacity of the C – V curves in enhancement mode and increases the shift of the C – V curves towards positive values, i.e., produces a negative fixed charge (Fig. 2, Curves 4 and 5);

- if plasma pretreatment is used a change in the control voltage sweep direction changes the capacity in the C – V curves at the same control voltage. Figure 3 shows the C – V curve of a plasma pretreated structure (40 s exposure) for different sweep directions (direct and reverse): measured capacities are different for the same control voltage;

- with an increase in plasma treatment duration the free carrier concentration in the 2D gas channel decreased (Fig. 4, Table 2). For example after 200 s plasma treatment the carrier concentration was $(1-2) \cdot 10^{12}$ cm⁻² ($(1-2) \cdot 10^{18}$ cm⁻³);

- for plasma pretreatment duration of 25 to 200 s the I – V curves of dielectric/AlGaIn/GaN structures exhibited current oscillations (Table 2, Fig. 5).

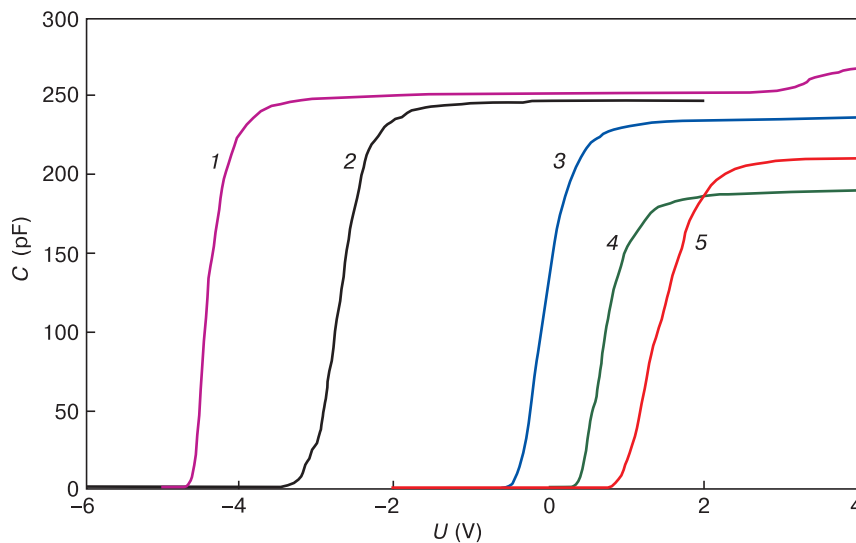


Figure 2. C – V curves of SiN_x/AlGaIn/GaN heterostructures with Si : N = (40 : 60)%. (1) SiN_x film deposited without nitrogen plasma treatment and (3–5) with additional plasma treatment for (3) 50, (4) 100 and (5) 200 s.

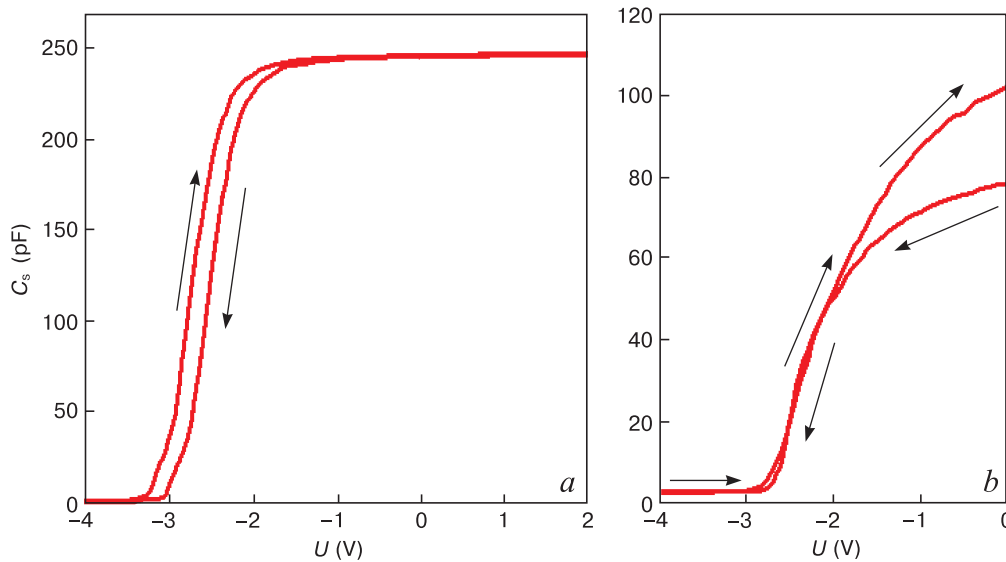


Figure 3. C–V curves of SiN_x/AlGaIn/GaN heterostructures synthesized with additional 20 s plasma treatment during SiN_x film deposition for different curve sections: (a) hysteresis and (b) recovery. Arrows show sweep direction.

Analysis of current oscillation evolution with an increase in direct surface plasma pretreatment duration showed a nonmonotonic pattern of oscillation period and amplitude variation, the general tendency being decreasing oscillation period and increasing oscillation amplitude with an increase in plasma pretreatment duration (Table 2, Fig. 5). For long pretreatment (200 s) the C–V curves are almost absent at 1 MHz ($C = 10$ pF).

Analysis of the effect of HF bias during direct surface plasma pretreatment before PECVD showed that biasing dramatically increases the length of the I–V curve section in which current oscillations occur, i.e., increases the number of oscillations (Table 2).

Thus taking into account earlier data [9] it can be concluded that the fixed charge in the PECVD SiN_x/AlGaIn/GaN

films containing 60% N and 40% Si is the same as in films with higher (above 5%) oxygen content (Table 1). This most likely indicates that synthesis of these films in dielectric/AlGaIn/GaN structures generates a system of elastic stresses with close magnitudes and similar sign of stress in the AlGaIn layer and hence these two examples of dielectric/AlGaIn/GaN systems are similar from the viewpoint of piezoelectric charge forming at the AlGaIn/GaN interface. Furthermore a common feature of these PECVD processes is the presence of current oscillations in the I–V curves of the structures.

As noted above the I–V curves of the initial AlGaIn/GaN heterostructures and the dielectric/AlGaIn/GaN structures were studied in many works [16, 17] dealing with HEMT technology. Little attention however was paid in these works to low current ranges of the I–V curves. Earlier [9] and in this work we found unexpected nonmonotonic leakage current vs control voltage dependences. Experiments showed that the occurrence and pattern (amplitude, period and control voltage range length) of these oscillations are controlled by the parameters of the dielectric/AlGaIn interface synthesis process and heterostructure surface preparation to PECVD.

No current oscillations were observed for SiN_x films synthesized by atomic layer deposition (ALD), and even for PECVD SiN_x films current oscillations were only the case for high nitrogen or oxygen contents (Table 1). Thus the experimental results suggest that the key condition for the occurrence of current oscillations in the I–V curves of nitrogen or oxygen rich films is plasma pretreatment before synthesis. Furthermore studies showed that an increase in the duration of additional direct nitrogen plasma pretreatment of heterostructure surface changes the current oscillation pattern, i.e., increases the oscillation amplitude and slightly reduces the oscillation period (Table 2, Fig. 5).

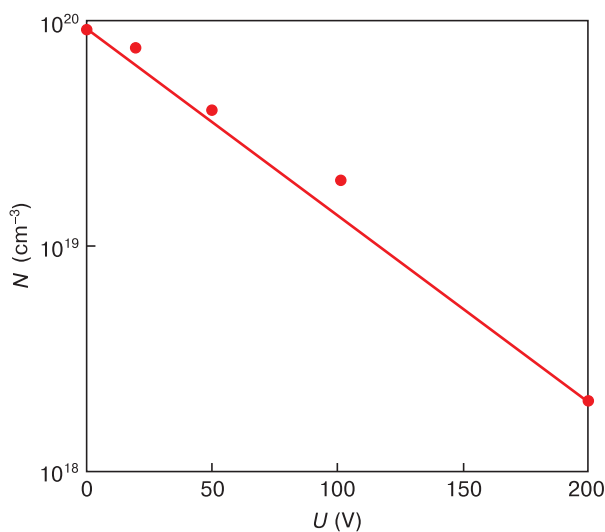


Figure 4. Carrier concentration in 2DEG for SiN_x/AlGaIn/GaN structures as a function of additional plasma treatment duration of heterostructure surface during SiN_x film deposition.

Table 2. Electrical parameters of SiN_x/AlGaIn/GaN structures

Structure No.	Plasma treatment duration (s)	Measured capacity (pF)		Carrier concentration in 2DEG			Oscillations		Cutoff voltage (V)
		Before SiN _x deposition	After SiN _x deposition	Hall effect (10 ¹² cm ⁻²)	C-V measurements (10 ¹⁹ cm ⁻³)	Period (V)	Amplitude (rel.u.)	Oscillation section length (ΔV)	
1	0	1550	246	11	8.5–9.0	0.18	1	3.5	–6 ÷ –6.5
2	25	1400	249	8.5–9.0	7.5	0.16	1.2	2.8	–0.5 ÷ –0.8
3	50	1500	192	8.4	4.0	0.14	1.5	4.5	0.5
4	100	1499	219	6.54	2.5	0.12	1	2.5	1
5	200	1500	–	1–2	0.21	0.18	0.2	0.7	–
6	159 (with HF bias)	1200–1300	250 (at f=10 kHz)	–	0.3–0.5	0.16	0.6	6	–2 ÷ –1

Note. The SiN_x layer was deposited with different-duration additional nitrogen plasma pretreatment.

As noted above, a flux of positive ions (nitrogen, silicon, hydrogen and respective radicals) is directed to the surface of a heterostructure during standard PECVD [19]. Their energy is sufficient for the activation of chemical processes, desorption of volatile molecules from the AlGaIn layer surface but this energy is below the threshold one required for ion beam sputtering of the substrate material [10, 18]. The results presented above suggest that PECVD with an excess of chemically active elements such as oxygen and nitrogen provides for higher formation rate of their compounds. Transmission electron microscopic study [19] of the atomic structure of interface layers synthesized by low pressure chemical vapor deposition (LPCVD) for SiN_x on GaN showed that the interface layers have a crystalline structure with different boundary atomic configurations depending on deposition conditions, and the main portion of the amorphous dielectric layer forms later. The initial interface layer configuration may further change due to stress relaxation. It should also be borne in mind that the structure forming at the layer interface depends on the condition of the surface AlGaIn layer, e.g. the presence and thickness of the natural oxide layer on its surface [19]. On the other hand our studies also showed that the occurrence and pattern of current oscillations correlate with the condition of the forming dielectric/AlGaIn interface. Furthermore one should take into account that unlike earlier results [19] where boundary configurations for SiN_x/GaN were dealt with, we studied the SiN_x/AlGaIn interface where aluminum atoms increase the possibility of oxygen bond formation. As regards nitrogen, it forms bonds both with aluminum and gallium since nitrogen is ionized in the plasma and becomes chemically active. Taking into account the small thickness of the AlGaIn layer (20–25 nm), oxygen or nitrogen excess at the dielectric/AlGaIn interface during deposition may probably cause drain of these active impurities from the dielectric interface toward the AlGaIn/GaN interface and saturation of the interface layer with these impurities. As shown earlier [20] redistribution of impurities like oxygen, nitrogen and carbon may occur in AlGaIn/GaN heterostructures under specific conditions.

We therefore assume that in the cases in question a defective region may form in the vicinity of the GaN interface of the AlGaIn layer, this region being enriched with oxygen, nitrogen, aluminum, gallium interstitial atoms and their complexes. This in turn may lead to the formation of high density electrically active boundary states and a subband in the buffer layer near the AlGaIn/GaN interface. Comparison between the C–V and I–V curves of the structure showed that current oscillations occur at voltages for which the electric field already affects the AlGaIn region near the AlGaIn/GaN interface (Fig. 1 c). Analysis of DC current oscillations for specimens deposited without plasma pretreatment showed that the carrier concentration in the 2D gas channel of the SiN_x/AlGaIn/GaN structure remains the same as in the initial AlGaIn/GaN heterostructure before deposition (Table 2).

Current oscillations in the I–V curves of the heterostructures were also observed in a number of experiments. For example current oscillations were observed in the I–V curves for quantum well heterostructures and for GaAs films with different doping levels [21]. This was attributed to the formation of static and migrating acoustic domains in the structures. The same mechanism of current oscillations was suggested [22] for AlGaIn/GaN heterostructures. Photocurrent oscillations in AlGaIn/GaN heterostructures were observed to depend on the composition of the dielectric on the AlGaIn surface [12], and the oscillations were attributed to the formation of specific composition defects. In our opinion the occurrence of the oscillation current component in the AlGaIn/GaN heterostructure for certain structure surface treatment modes is caused by AlGaIn subband formation in the vicinity of the AlGaIn/GaN interface, i.e., tunneling of electron/hole pairs through virtual quantum well states forming in this region of the structure [23, 24].

Thus some PECVD modes may favor the formation of high concentration impurity defects in the AlGaIn layer bulk without strongly changing the dielectric properties of this layer. As a result, according to C–V curve measurements, the free carrier concentration in the channel and the channel location relative to the heterostructure surface after dielectric deposition do not change in

comparison with the initial heterostructure before deposition (Table 2).

In our opinion, the understanding of the processes occurring during PECVD of SiN_x dielectric films on the surface of AlGaN/GaN heterostructures would be improved by experiments with different-duration direct nitrogen plasma treatment of heterostructure surfaces before monosilane supply. As noted above direct nitrogen plasma treatment of structure surfaces in standard plasmachemical processes cannot cause surface material sputtering due to low nitrogen particle energy (within 30 eV) but as shown earlier in a number of works [13–15] it may cause active nitrogenization of the material. An increase

in plasma treatment duration used for this process does not increase the penetration depth of nitrogen atoms into the material because the depth of the nitrogen-rich defect region in the AlGaN buffer layer does not increase, only the nitrogen atom saturation of this AlGaN layer grows [13, 25]. This is confirmed by our experimental results: for a 25 s plasma treatment duration nitrogenization of the surface region in the layer AlGaN produces a nitrogen-rich defective surface region which may change the elastic properties of the AlGaN/GaN interface. The elastic stress evolution pattern and hence piezopolarization of the AlGaN/GaN structure undergo such a dramatic change that the positive fixed charge is eliminated almost

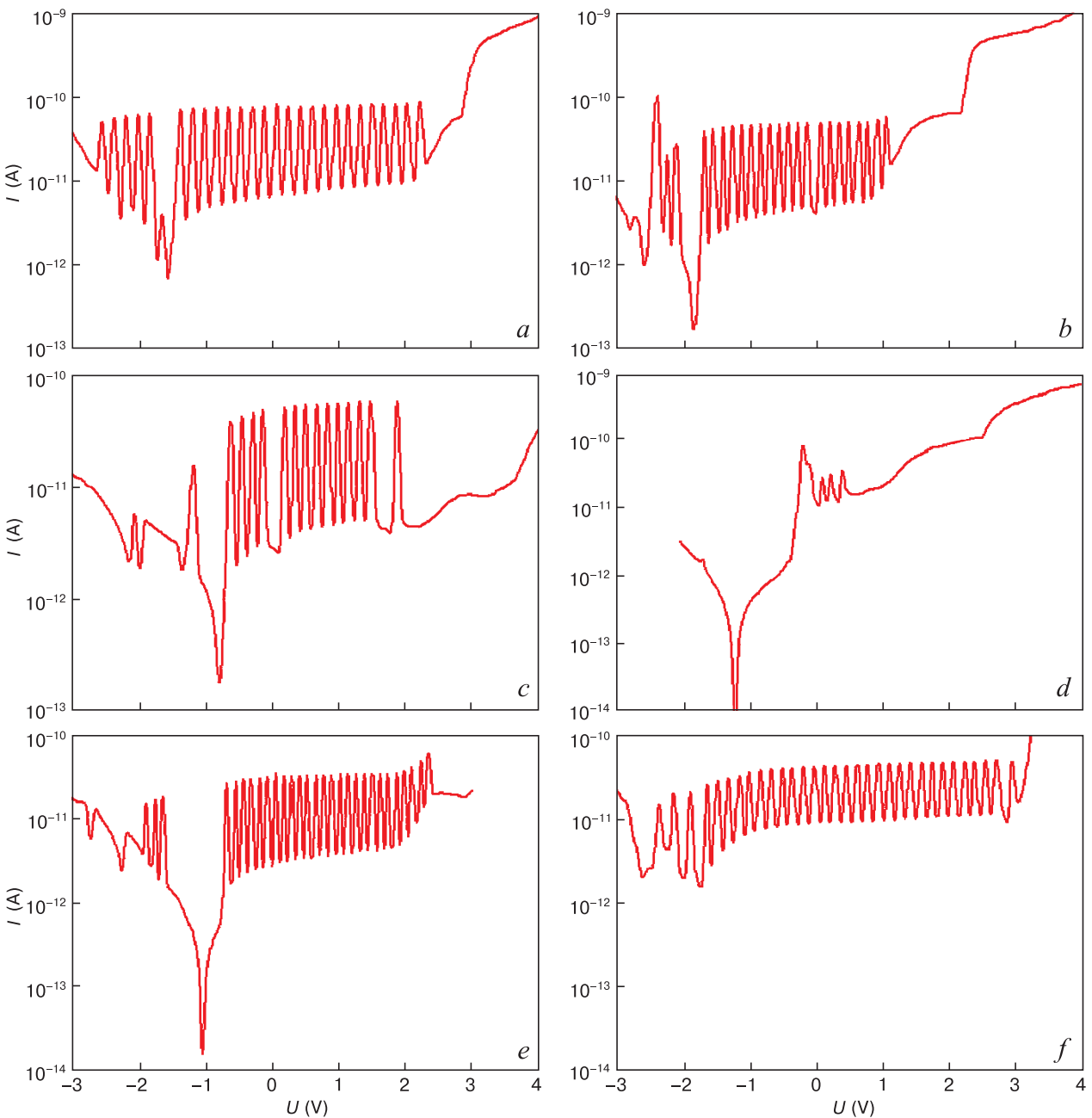


Figure 5. I–V curve sections with current oscillations for $\text{SiN}_x/\text{AlGaN}/\text{GaN}$ structures, SiN_x (a) without additional plasma treatment and (b–f) with additional plasma treatment in different modes: (b–e) plasma treatment for 25, 50, 100 and 200 s, respectively and (f) plasma treatment with HF bias.

completely in this system: U_{cutoff} tends to zero (Fig. 2, Curve 3). As can be seen from Fig. 2 (Curves 4 and 5), further increase in plasma treatment duration generates a negative fixed charge in the dielectric/AlGaIn/GaN structure due to an increase in the nitrogen saturation of the defect region in the buffer layer: U_{cutoff} shifts towards positive values. The capacity measured for direct sweep changes from 250 to 180 pF for almost the same C–V curve slope in the depletion to enhancement transition curve section. The carrier concentration in the 2D gas channel, e.g. for a 100 s plasma treatment duration, decreases by an order of magnitude (Fig. 4) and further increase in plasma treatment duration to 200 s reduces it to $(6\text{--}7) \cdot 10^{17} \text{ cm}^{-3}$. Furthermore direct plasma treatment of heterostructure surface does not lead to recovery of the measured capacity (Fig. 3 b) upon change in the control voltage sweep direction, and hysteresis occurs (Fig. 3 a). We believe this indicates that the case in hand involves not only simple nitridization of part of the buffer layer but also the formation of electrically active defects whose nature requires further investigation.

The role of plasma treatment in the formation of current oscillations is confirmed by an experiment with HF biasing of the heterostructure during plasma treatment of its surface. The efficiency of plasma treatment increases significantly due to acceleration of nitrogen particle flux toward the surface (the plasma particle energy is far above 50 eV). As can be seen from Table 2 this noticeably increases the number of oscillations (Fig. 5).

Thus the results of this work demonstrate the complexity of the impact from PECVD processes to heterostructures and the importance of surface preparation for this process, as well as how minor changes or negligence in technology can affect the electrical properties of growing AlGaIn/GaN structures with passivating layers. Furthermore in our opinion the occurrence of current oscillations despite the very low leakage current may cause quite unexpected phenomena during transistor operation, e.g. low-frequency noise.

4. Conclusion

C–V and I–V curve measurements for PECVD SiN_x/AlGaIn/GaN structures synthesized with various process options show that structures with SiN_x films containing 60% nitrogen and 40% silicon or oxygen rich ones (5–8%) exhibit leakage current oscillations in the I–V curves. Experimental evidence is reported proving that the parameters of these oscillations depend on heterostructure surface plasma treatment options. Explanations are provided as to possible origins of these oscillations. Additional direct nitrogen plasma treatment of AlGaIn/GaN heterostructure surfaces for different durations allows one to control the magnitude and sign of fixed charge in the SiN_x/AlGaIn/GaN system and change the free carrier concentration in the 2D gas channel in the AlGaIn/GaN system.

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